U. S. DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS JOURNAL OF RESEARCH

Vol. 7 AUGUST, 1931 No. 2

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Bureau of Standards Journal of Research

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BUREAU OF STANDARDS JOURNAL OF RESEARCH

August, 1931
Vol. 7, No. 2

UNITED STATES
GOVERNMENT PRINTING OFFICE
WASHINGTON : 1931

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A NEW APPARATUS FOR PREPARING Ra B+C SOURCES

By L. F. Curtiss

ABSTRACT

A simple glass apparatus is described which may be used to serve the purpose of an "emanation pipette" but which eliminates two objectionable features present in the usual forms of this apparatus. Mercury does not come in contact with rubber tubing, since no rubber tubing is used. A further improvement is accomplished by avoiding the necessity for passing mercury through stopcocks.

The preparation of radium B+C sources is usually done by some form of emanation pipette, such as is shown in Figure 1. This consists of a 2-way stopcock with a large glass barrel, *B*, below into which mercury can be forced by a reservoir, *R*, attached to the lower end by rubber tubing, *T*. It is operated first as a pump to exhaust the portion sealed on at *A* where the source is to be prepared. The purified emanation is introduced through *C* into *B* and, by reversing the stopcock, it is forced into *A*. This form of apparatus has two very serious defects which render it unsatisfactory for continuous operation. The rubber tubing soon introduces impurities into the mercury and causes an accumulation of "dirt" on the inside of the barrel which interferes with the efficiency of the device. A more serious trouble arises from the fact that mercury must be forced through the bores of the stopcock at certain stages of the operation of the apparatus. Even with the greatest care in greasing the stopcock, the mercury gradually becomes contaminated with the grease which, under the action of the radon, becomes hard enough to choke the bore of the capillary, especially if the usual rubber greases are used. A description is given below of a device which eliminates both of these difficulties, thereby making the preparation of active deposits from radon a much simpler and less tedious operation. It also possesses the advantage that practically no radon is lost in the preparation of a source by being trapped by dirt which has accumulated in the apparatus. This is very important where it is desired to prepare a number of sources from the same radon.

The essential details of the apparatus, which is entirely of glass, are shown to scale in Figure 2. It consists of two principal parts, one in which the source is actually prepared, and an auxiliary part which is an ordinary Toepler pump arranged to be operated by compressed air. The various parts and their functions can best be described by outlining the method of using the apparatus when preparing a source.

The steel reservoir, *R*, is filled with mercury covering the open end of the capillary at *D*. To prepare the apparatus for use, the stopcock, S_1, is closed and S_2 is opened and the apparatus exhausted by forcing mercury, contained in the bulb *B*, repeatedly into the barrel of the Toepler pump, *T*. The check valve, *G*, has a ground seat which

prevents mercury rising above *G*. The method of using compressed air for operating a Toepler pump has already been described elsewhere.[1] It has been used for several years in the radium laboratory at the National Bureau of Standards, and has proven very satisfactory. Apparently there is no danger of breaking glass parts with pressures required for this purpose, since this laboratory has had no trouble of this kind. As can

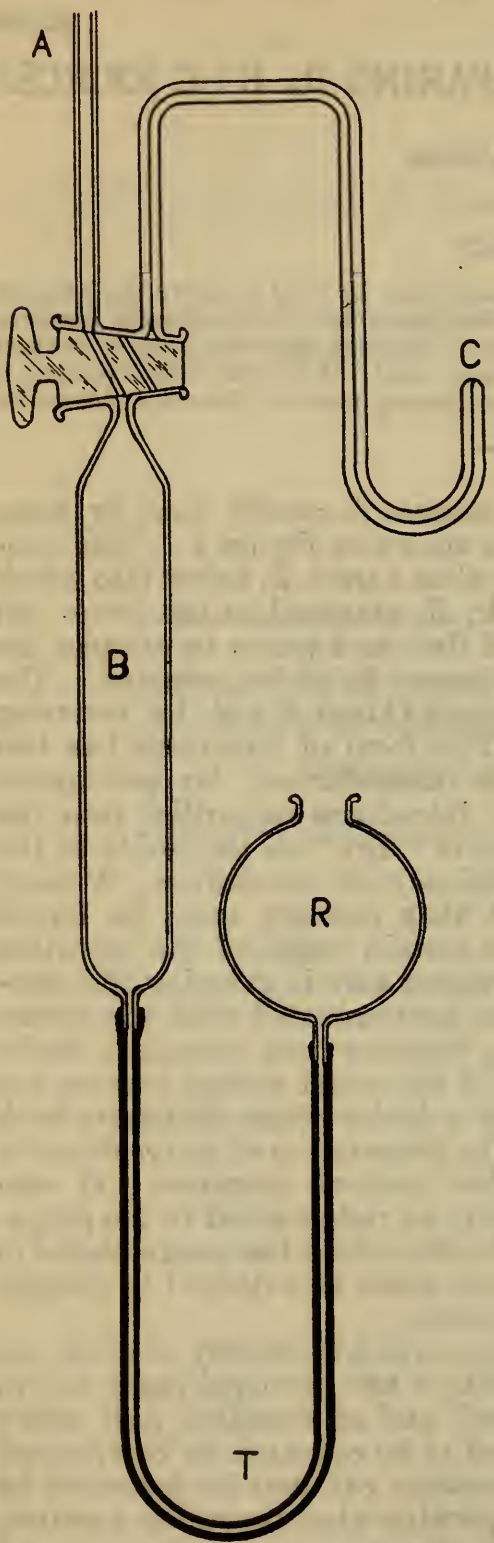

FIGURE 1.—*Old type emanation pipette*

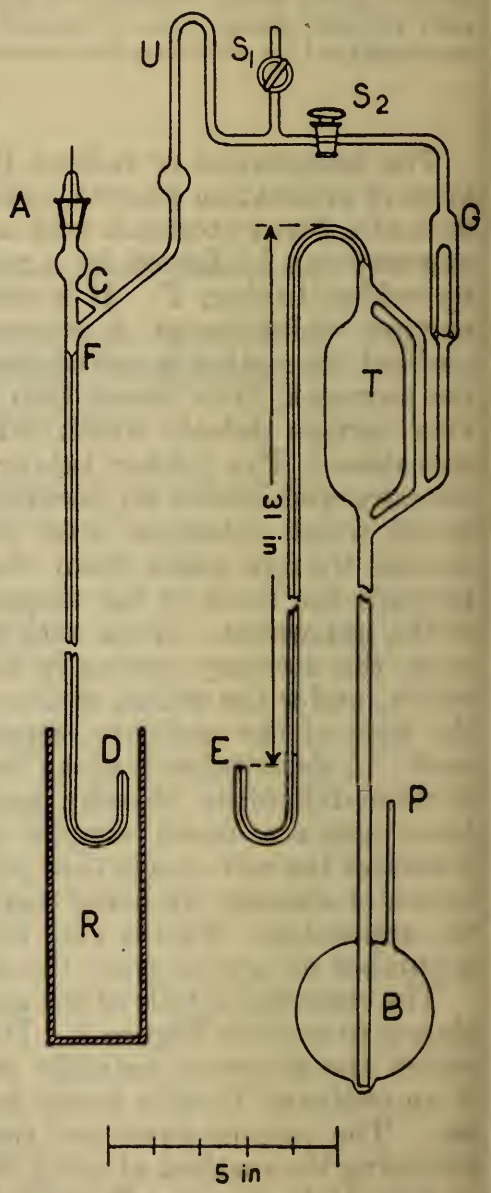

FIGURE 2.—*All-glass apparatus for preparing Ra B+C sources*

be seen it effectually eliminates the contact of mercury with rubber and makes a rapid operation of the pump possible and safe.

[1] L. F. Curtiss, J. Opt. Soc. Am. & Rev. Sci. Inst., **17**, p. 77; 1928.

The mercury reservoir, R, is attached to a counterpoise, not shown, which enables the position of the reservoir to be adjusted and consequently the level of the mercury column in the capillary above it. The next step in the operation is to adjust the level of the mercury in the capillary until it is at some point slightly below F. Then with the purified emanation at hand, stored in the usual way in an inverted test tube over mercury, the level of the mercury is raised by suddenly shifting the reservoir, R, upward about 2 inches. As the mercury rises to fill the branch tube at C, the radon, which is always of small volume, is introduced at D in the same way one would introduce it into an emanation pipette, simply slipping the inverted tube over the end of D. The radon will now rise quickly in the capillary and escape above the mercury column into the space below the stopper at A. Since the mercury level is by this time above the branch tube at C, any bubbles of radon which may rise up the side tube are caught by C and returned to the main volume. The mercury level is now adjusted to give a small volume of gas below the stopper which carries the metal button for receiving the active deposit. The usual voltage connections are made to the wire through the stopper, and by means of the mercury in the reservoir R.

When the exposure has been completed the radon is recovered in the following way. The reservoir R is lowered until the mercury column is again below the side tube at F. This allows the radon to expand into the evacuated tubes leading to the Toepler pump. An inverted test tube filled with mercury is placed over E, which is provided with a mercury reservoir similar to R, and the Toepler pump is operated until all radon has been pumped over into the test tube. Then, in order to remove the stopper, A, and to recover the source, air is admitted by the stopcock, S_1, while S_2 is closed. If desired both stopcocks may be eliminated, since S_2 only permits air to be admitted to the exposing tube without entering the Toepler pump so that the pump is a little easier to start when next used. The function of S_1 can be equally well performed, though less conveniently by a side tube drawn down to smaller diameter so that it may be cracked off to admit air.

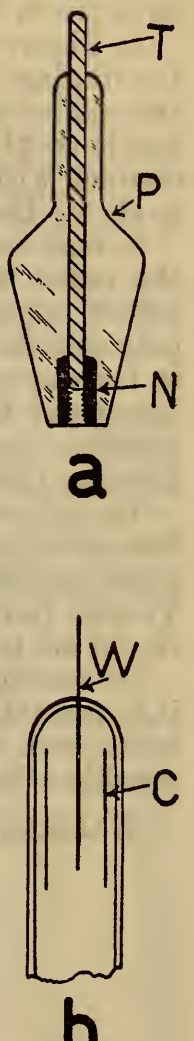

FIGURE 3.—*Pyrex stopper*

(*a*) Details of pyrex stopper; (*b*) arrangement for preparing active deposit on wire.

The two small bulbs shown in the glass tubing are important. The one in the vertical column prevents radon being trapped under a short column of mercury when rising into A. In the same way the bulb below U prevents a bubble of radon from carrying mercury up over the tube U which is purposely made high enough so that a solid column of mercury can not rise over it for any possible position of the reservoir, R.

The apparatus may be constructed either of soft glass or pyrex. For activating metal buttons a pyrex stopper, which, of course, can be ground into apparatus made of soft glass, is shown in Figure 3 (*a*). Since the tungsten wire, T, can not be threaded, a nickel bushing,

N, is held by a drive fit to the lower end which is tapped for a small screw. This combination is sealed into the pyrex as shown, the tungsten making a vacuum-tight seal to the glass. Metal buttons of various shapes, provided with a short threaded boss, may then be screwed into the nickel socket so that they are tight against the lower surface of the stopper, which should be ground flat. There are then no pockets in which radon can accumulate where it would not be effective in forming a deposit on the surface of the metal button.

In Figure 3 (*b*) is shown the usual method of preparing wire sources. If soft glass is used, the wire, *W*, is of platinum and is surrounded by a thin nickel or steel cylinder, *C*, which is not in contact with the wire, but is so placed that the mercury column can be raised to come in contact with it. Thus the nickel cylinder forms the positive electrode for the voltage used in preparing the active deposit.

It may be well to call attention to the fact that for best operation the radon should be freed as well as possible from the other gases before introducing it into the apparatus, since this apparatus can only handle small volumes of gas. A fairly high "purity" of radon is necessary in any case to get strong sources. It is even more important that the radon be carefully dried, since the presence of moisture causes the mercury to adhere to the glass and prevents keeping the glass and mercury surfaces clean.

By applying liquid air to a piece of cotton wool, wrapped about the tubing at *U*, it is possible to condense the radon and remove any gases noncondensable at liquid air temperatures by means of the Toepler pump. Then the radon may be pumped out by allowing the liquid air to evaporate.

By sealing on small diameter tubing at *A* in place of the stopper, it is possible to modify the apparatus so that it may be used to fill bulbs with radon, provided the reservoir, *R*, is designed to permit a considerable movement of the level of the mercury column.

Washington, May 1, 1931.